

Development of Stud-Pull Apparatus for Interface Strength Characterization

Honors Thesis

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Abstract

Stud-pull testing is used to measure the pull-off strength, or adhesion, of a coating to a rigid substrate. A stud, made of some rigid material, is connected to the coated substrate with the strongest available adhesive and once the adhesive has cured, the stud is separated from the substrate. It is important that the stud-adhesive interface and the adhesive-coating interface are stronger than the coating-substrate interface because failure will occur at the weakest interface in the system. The adhesion of the coating is determined by measuring the greatest perpendicular tensile force that can be applied to the coated area before failure occurs. The pull-off strength will depend on both material and instrumental parameters [14].

A stud-pull apparatus requires a moving actuator that separates the stud from the substrate and a fixed base that holds the substrate in place. The actuator and accompanying load cell (used to measure the force) were provided for by an 800LE series load frame from TestResources. The remaining components were designed to accommodate 1" diameter circular substrates with seven coating islands of 3/16" diameter. It is desirable to have a self-aligning stud such that the stud is perpendicular to the specimen regardless of the substrate's surface features. This was accomplished by inserting an inline ball joint between the load cell and the stud, giving the stud the freedom to pivot and twist in all directions. The end of the stud which contacted the substrate was made of 1/8" diameter aluminum. This diameter was chosen because it is large enough for the stud to be rigid in tension and it is small enough for multiple tests to be conducted on a single 1" diameter substrate. The fixed base of the apparatus consisted of a 2" diameter aluminum "table" that fastened to the base of the load frame and a 2" diameter retaining ring that fastened over the table. The substrate was centered on the table and the retaining ring clamped over the substrate, effectively holding the substrate in a stable position. During test

preparation, epoxy was applied to the end of the stud and the stud was inserted through the center of the retaining ring to reach the coated area of the substrate. When testing occurred, the stud was raised through the center of the retaining ring by the load frame actuator and the load, position, and time at failure were noted.

The first 22 stud-pull tests were conducted with uncoated aluminum substrates for the purpose of testing the pull-off strength of the epoxy. Loctite E-120HP Ultra-Strength epoxy was chosen for its rated tensile strength of 5900 psi, which was the highest available strength. The initial tests varied cure time of the epoxy, separation rate of the stud from the substrate, and surface roughness of the aluminum substrate. The next 14 tests were conducted with painted aluminum substrates. A template was designed and machined to easily spray-paint square aluminum test pieces with seven islands of 7/32" diameter. The Aervoe Rust Proofing Paint was chosen for its availability in the lab and for its useful applications as a common interface. Two painted substrates with seven islands each were tested: one with a smooth surface, the other with a rough surface. The effects of cure time and surface roughness were evaluated.

Now that the stud-pull apparatus has been thoroughly tested under various conditions and after numerous complications, it can be used to test more significant specimens. The initial motivation for this project was to evaluate the anode-electrolyte interface strength of specimens that have applications in solid oxide fuel cells (SOFC's), but no SOFC specimens could be tested due to their high cost and difficulty to obtain. However, it was imperative that the stud-pull apparatus be tested extensively before real specimens could be used. Any loss of data from an SOFC specimen would be a very costly error.

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1. Introduction and Motivation

The motivation for developing stud-pull apparatus stems from the need to characterize degradation in solid oxide fuel cells (SOFC's). All fuel cells operate on the principle of chemically combining hydrogen and oxygen to form electricity, water, and other products. This principle is essentially the opposite of the electrolysis of water. Many of these single cells are connected together in series to form a fuel cell stack, which can generate electricity on the order of tens to hundreds of kilowatts [11]. Figure 1 is a schematic that shows the operating principle for a single fuel cell.

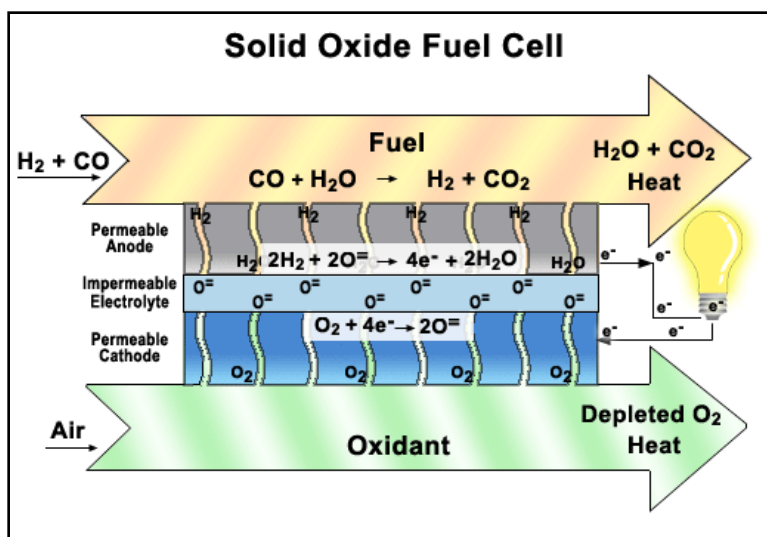


Figure 1: A Single Fuel Cell in an SOFC Stack [1]

There are various types of fuel cells with very different materials and operating characteristics, including phosphoric acid fuel cells (PAFC), proton exchange membrane fuel cells (PEMFC), molten carbonate fuel cells (MCFC), and alkaline fuel cells (AFC). PAFC's allow impure hydrogen as a fuel source, utilize liquid phosphoric acid as the electrolyte, operate at 450°F, and can achieve more than 40% efficiency. PEM's have a polymer electrolyte

membrane, operate around 175°F, and are very compact; however, they are easily poisoned by impurities such as carbon monoxide. MCFC's have an electrolyte composed of a molten carbonate salt mixture suspended in a porous and chemically inert matrix. They operate at 1200°F, require carbon dioxide and oxygen at the cathode, and tolerate a wide variety of fuel sources: hydrogen, carbon monoxide, natural gas, propane, and more. AFC's have been used on the NASA space missions as a source of electricity and drinking water and they operate at about 160°F. The electrolyte is potassium hydroxide and they are easily contaminated by carbon, so pure hydrogen and oxygen are required [7].

An SOFC works by passing hydrogen-rich gaseous fuel over an anode and air over a cathode with an electrolyte in between. When an oxygen molecule contacts the interface between the cathode and electrolyte, it gains four electrons from the cathode and splits into two oxygen ions. The oxygen ions conduct through the electrolyte and react with hydrogen (and some carbon monoxide) at the anode interface to produce water, carbon dioxide, heat, and electricity [12]. The electrons are transferred to an external circuit and back to the cathode, creating useful electrical energy.

Since materials for SOFC's are chosen in large part for their electrochemical performance and thermal stability, there is limited opportunity to make significant changes to component materials [2]. SOFC electrolytes require high oxygen ion conductivity, so they are almost always made of yttrium-stabilized zirconia (YSZ). Typical anodes consist of a composite of nickel-gadolinium doped cerium oxide and nickel-yttria stabilized zirconia (Ni-GDC/Ni-YSZ); a typical cathode is a Lanthanum strontium manganite (LSM) multilayer [17]. In order to achieve sufficiently high conductivity in the component materials, SOFC's have to run at operating temperatures upwards of 800°C. The primary benefit to the high operating temperature is fuel

flexibility and tolerance to fuel impurities. Unfortunately, higher operating temperatures increase the thermal expansion mismatches of the component materials, causing high thermal stresses and material degradation over time [5]. A scanning electron microscope image of the component interfaces in an SOFC is shown in Figure 2.

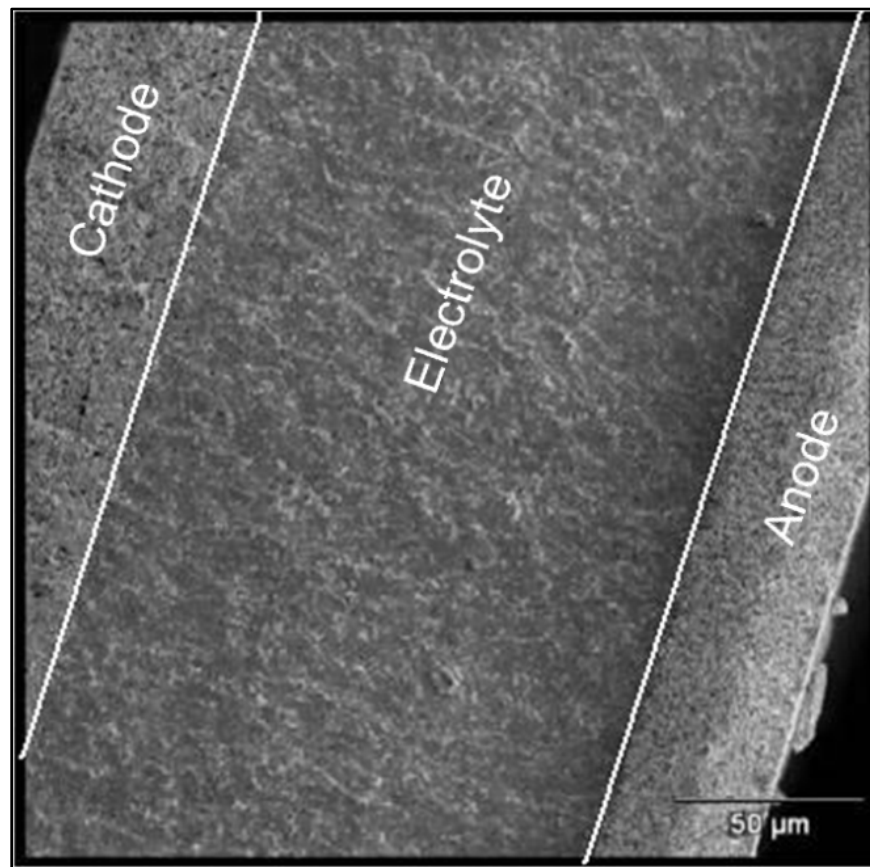


Figure 2: Cross Section of an SOFC Cathode, Electrolyte, and Anode from a Scanning Electron Microscope Image [13]

The degradation and failure of SOFC's typically occur following the shutdown and startup phases. Thermal expansion coefficient mismatches between ceramic materials with low toughness and thermal conductivity can result in critical failure if the rate of cooling and heating

is more than about 1-3°C/min [17]. One way of solving the problems associated with thermal degradation is to improve the mechanical strength of the components, but even after strengthening, the electrode interfaces still experience microstructure changes and delamination during thermal cycling. The purpose of this project is to develop apparatus that will enable investigation of the mechanical strength of the anode-electrolyte interface.

2. Review of Interface Strength Characterization Methods

Interface strength testing is used to determine the strength of adhesion between a coating and a substrate. These interface tests can be qualitative or quantitative, depending on the type of test and the desired result. Different types of tests include the bend test, peel test, indentation test, scratch test, and pull test. Bend tests give qualitative information for ductility and material soundness by bending a specimen to a specified angle [16]. Peel tests measure the strength required to pull apart a bonded surface and are useful in evaluating adhesives [8]. In an indentation test, a probe that is aligned normal to a sample is driven into the sample with increasing force until a preset value is reached. The probe is then relaxed partially or completely to generate a load vs. depth curve that can be used to measure material properties such as hardness and elastic modulus for nearly any type of material [3]. The scratch test is similar to the indentation test, where a diamond tip probe is aligned normal to a sample and a load is applied. In a scratch test, however, the probe is drawn across the material in constant, incremental, or increasing load. The normal force, tangential force, and depth at failure can be used to characterize surface properties [4]. The pull test is similar to the peel test, where the strength of an interface is tested by pulling the interface apart. This project utilizes the stud-pull test for its ability to quantify the strength of adhesion of a coating to a substrate. A quantitative

stud-pull test is position-controlled, where the stud is slowly separated from the test specimen such that the rate of stress does not exceed 1 MPa (150 psi/s), as specified in the ASTM D4541 standard for pull-off strength testers [14]. Failure occurs when a plug of material is detached within the system. The interface in which failure has occurred is noted and the pull-off strength is calculated based on the maximum load and the original stressed surface area. A generic stud-pull test setup is shown in Figure 3.

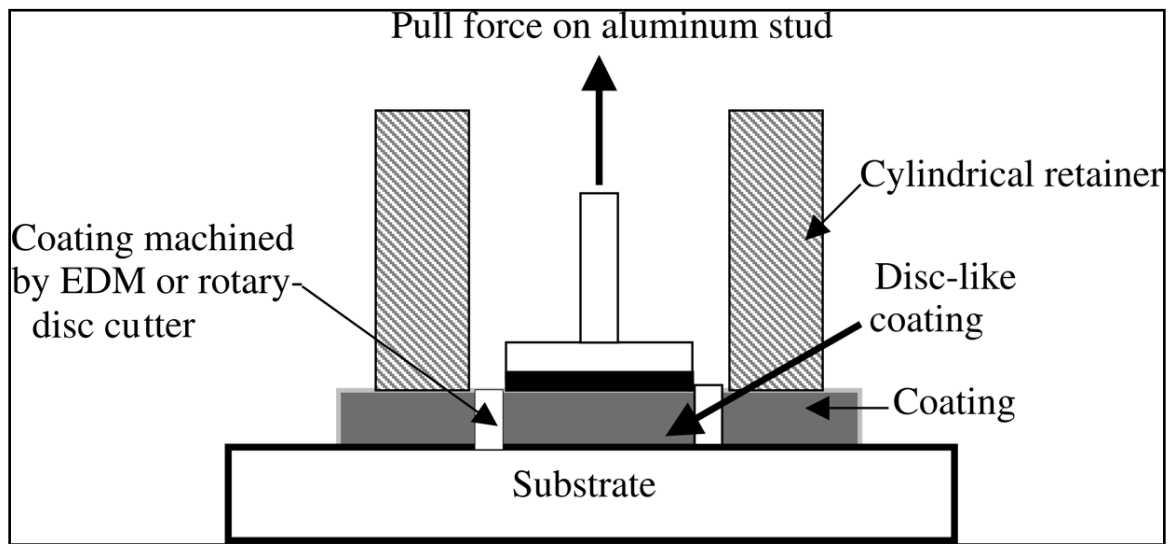


Figure 3: Schematic of a Stud-Pull Test [6]

The setup in Figure 3 features a cylindrical retainer and a coating area that has been machined around the diameter of the stud. Elmoursi and Patel performed four types of stud-pull tests with setups similar to the schematic shown in Figure 3. The first method had no retaining cylinder and the coating was not isolated. The second method incorporated the retaining cylinder. In the third method, the coating was machined by electric discharge machining (EDM) to match the coating diameter to the stud diameter. The fourth method was similar to the third but replaced EDM with a rotary-disc cutter. The conclusions they reached were that the first and

second method gave consistent measurements for adhesion as long as the coating thickness was less than 2.5% of the stud diameter. For thicker coatings, the third and fourth methods were recommended [6]. The ASTM standard also notes that scoring around the stud to isolate the coating violates the in-situ test criterion by altering the coating and can cause micro-cracking, which reduces adhesion [14].

The cylindrical retainer introduces a shear component on the coating along the direction of the cylindrical wall, which results in a higher pull-off force that is required for failure [6]. That is why the coating thickness is an important factor in the setup shown in Figure 3. Another factor that the cylinder introduces is a degree of bending when the substrate is in tension, regardless of whether or not the coating is isolated. This means that the rigidity of the substrate can affect the pull-off strength, where a thinner substrate of almost any material will experience a lesser pull-off strength than a thicker substrate [14]. A more effective method of separating the coating from the substrate without introducing bending would be to make the substrate, coating, adhesive, and stud at identical diameters. This would also help with accurately aligning the system, as error is introduced in the pull-off strength if the stud is not normal to the surface [14].

3. Experimental Setup

Once the apparatus shown in Figure 3 is in place, an actuator with accurate load measurement capabilities is required. All stud-pull tests were conducted using the 800LE series load frame from TestResources shown in Figure 4.



Figure 4: 800L Series Load Frame from TestResources

The load frame's accompanying software features real-time displays of the load and position of the load frame actuator (screen shots of the real-time displays can be found in the Appendix). The software enables the user to program the actuator to follow specific motions. Various data acquisition options are also available, and data can be saved to Microsoft Excel spreadsheets for subsequent analysis. The values of interest include the time, displacement, and load between startup and failure.

The experimental setup is shown in Figure 5 and Figure 6.

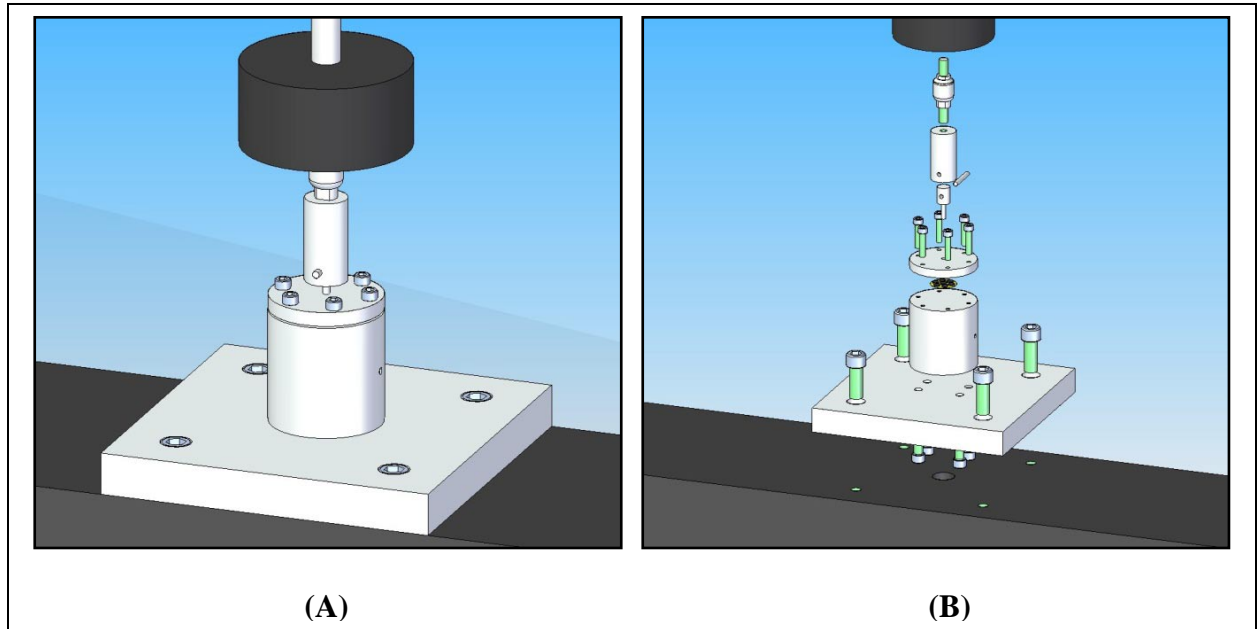


Figure 5: Solid Models of the Stud-Pull Apparatus: (A) Assembly and (B) Exploded View

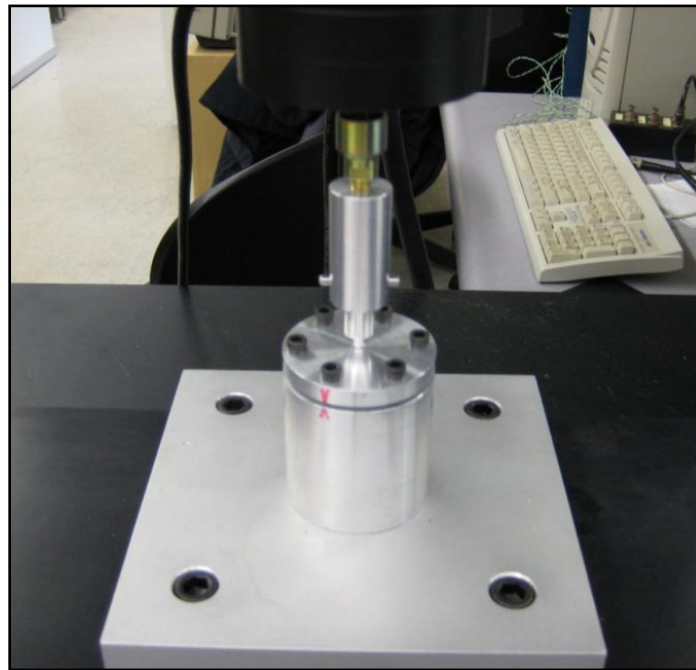


Figure 6: A Photograph of the Assembled Stud-Pull Apparatus

The experimental setup consists of two parts: a fixed base and a moving actuator. The fixed base consists of the load frame base, a base plate, a table, and a retaining ring. The base components are shown in Figure 7.

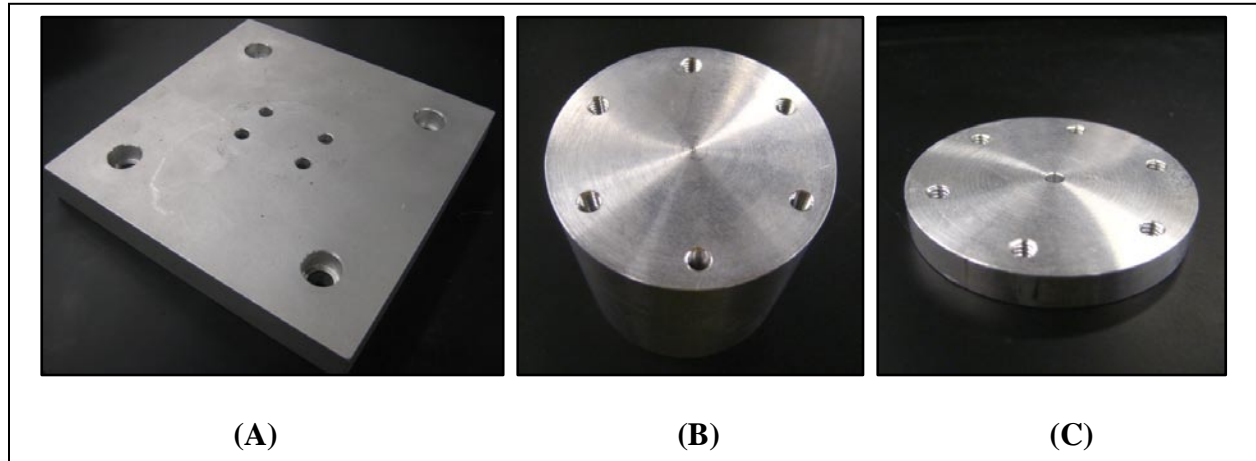


Figure 7: Fixed Base Components: (A) 6'' x 6'' x 0.75'' Base Plate, (B) 2'' dia. x 2'' Tall Table, and (C) 2'' dia. x 0.25'' Tall Retaining Ring

Four 3/8-16 UNC socket head cap screws (SHCS) fasten the base plate to the load frame and four 1/4-20 UNC SHCS connect the table to the base plate. The test specimen is centered on the table and a washer is centered on the test area as shown in Figure 8.

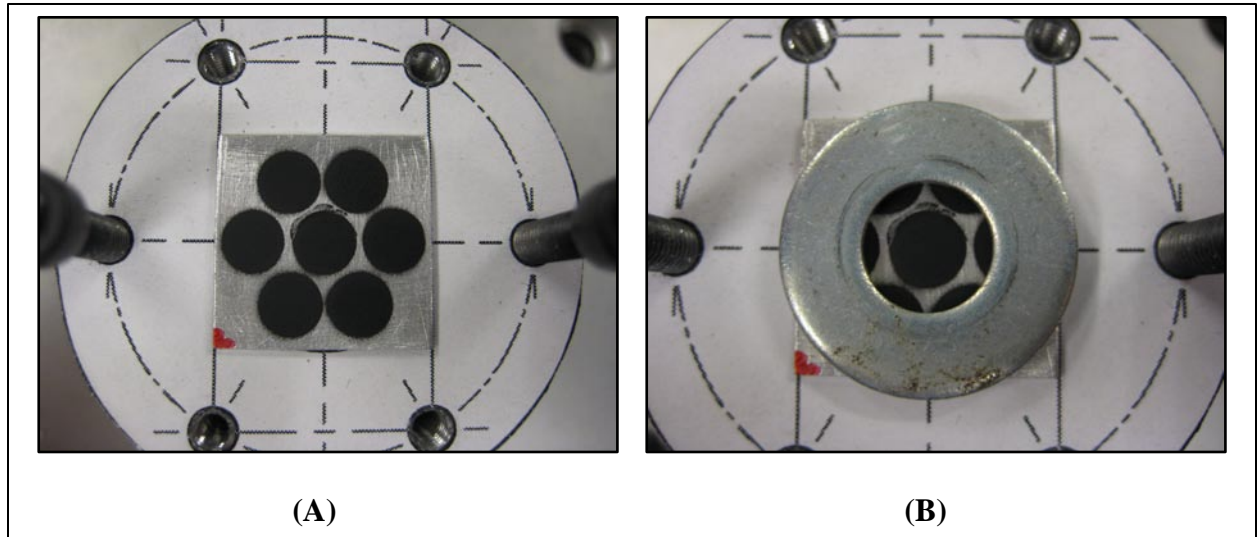


Figure 8: Test Preparation: (A) Specimen Centered on Table, (B) Washer on Specimen

A retaining ring is then placed over the washer and fastened to the table using six #8-32 UNC SHCS as shown in Figure 9.

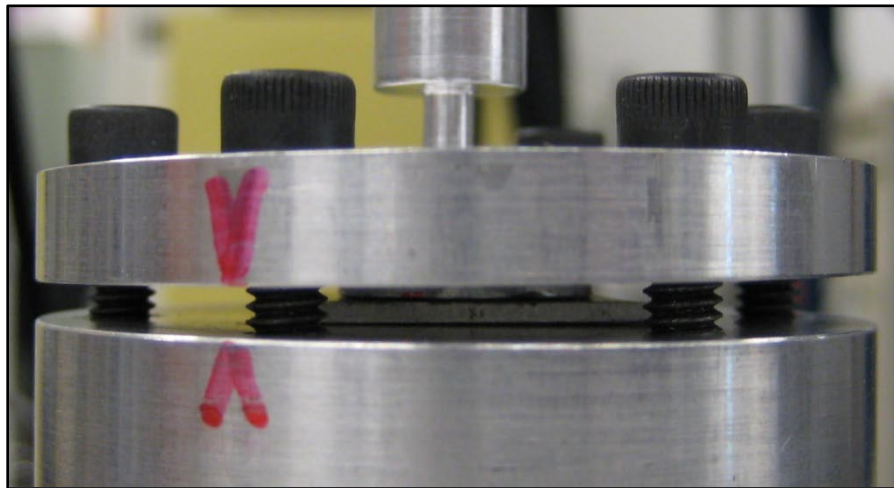


Figure 9: Retaining Ring Fastened over Washer and Test Specimen

Once all of the fixed base components have been securely fastened, the moving actuator components can be set up.

The black cylinder at the top of Figure 6 and Figure 10 is a 100 lb (450 N) load cell. The ball joint shown in Figure 10 allows the stud to rotate and twist in all directions. The ball joint also permits self-alignment of the stud to the test specimen instead of having a rigid stud with no maneuverability. During one of the stud-pull tests, the load frame actuator malfunctioned and compressed the stud into the test specimen, causing the 100 lb load cell to exceed its rated load capacity. This rendered the load cell inoperable. A 1000 lb load cell replaced the damaged load cell and a threaded adapter was implemented to connect the larger load cell to the inline ball joint.

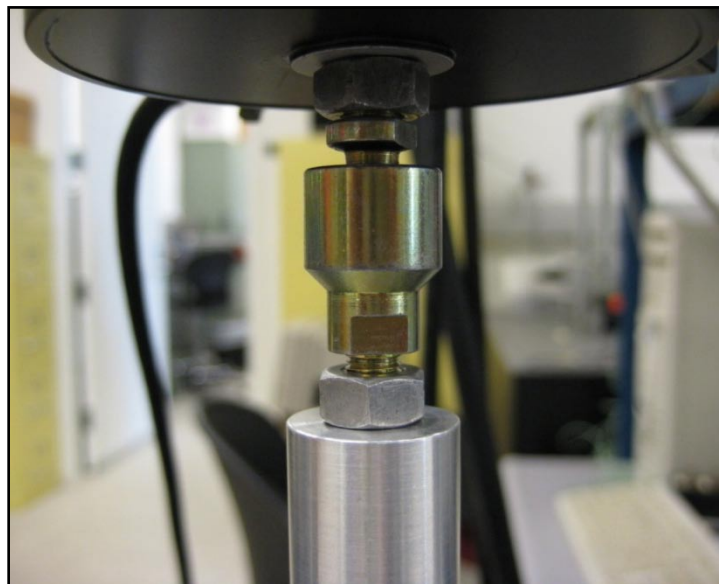


Figure 10: Inline Ball Joint Connecting Load Cell to Stud Holder

The nuts that fasten the inline ball joint between the load cell and the stud holder help to ensure that the stud remains perpendicular to the test specimen and helps with the positional repeatability from one experiment to the next because the stud holder must be removed and

cleaned after every stud-pull test. The remainder of the stud holder, as well as the stud that is pinned inside of the stud holder, is shown in Figure 11.

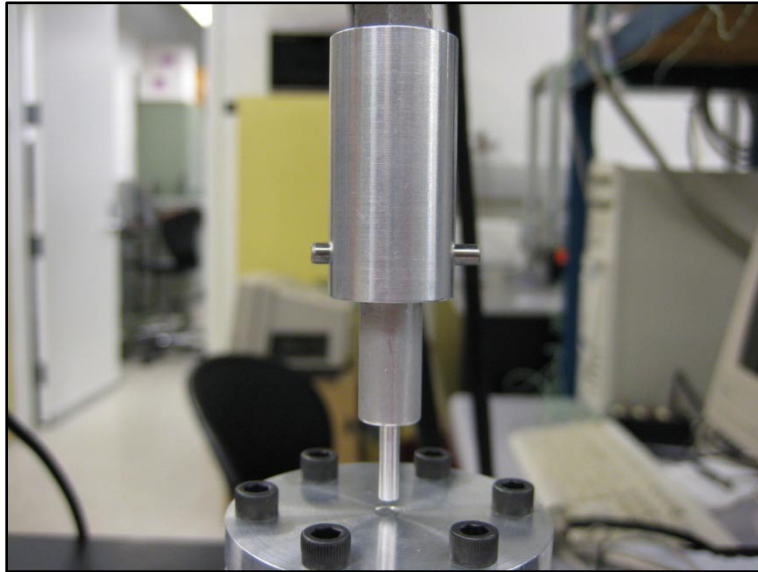


Figure 11: Stud Pinned Inside Stud Holder

When the top and bottom portions of the apparatus have been assembled, the final step to begin a test is to align the stud to the retaining ring's central hole. The diameter of the stud at the bottom is 0.125" and the retaining ring hole is 0.161". The larger retaining ring hole diameter is needed to give enough clearance so that the epoxy that is placed on the end of the stud does not touch the walls of the retaining ring's central hole. Before any epoxy is mixed, the stud is aligned to the central hole by hand and a simple code is programmed into the software that lowers the stud at 0.01 mm/s until a 1.0 N compressive load registers. Once there is a compressive load, the stud is in contact with the test specimen. This setpoint is noted and the stud is raised back out above the retaining ring. The epoxy is then mixed for several minutes and applied to the end of the stud, as shown in Figure 12.

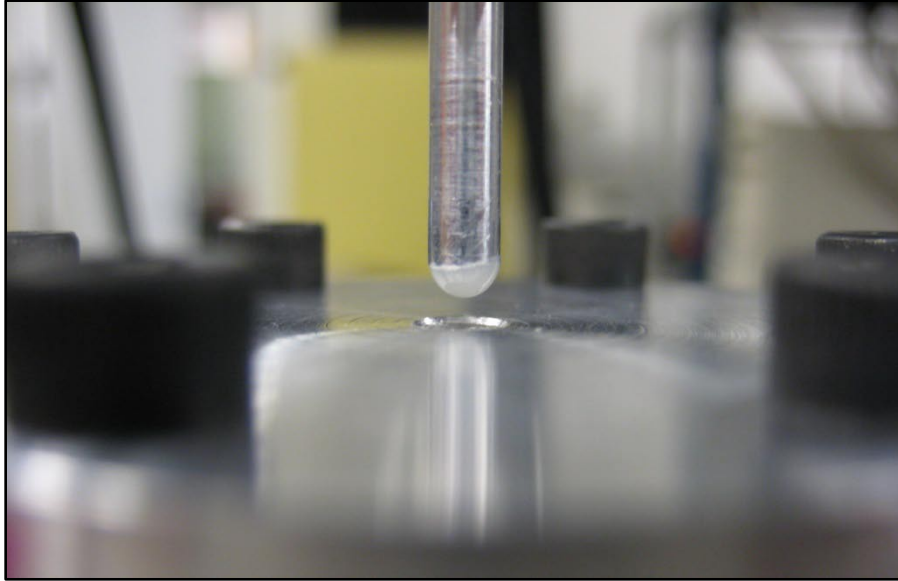


Figure 12: Stud with Epoxy during Test Preparation

The epoxy chosen was Loctite E-120HP Ultra Strength epoxy from McMaster-Carr. A full technical data sheet on the epoxy can be found in the Appendix. The epoxy has a 2:1 resin to hardener mix ratio and fully cures after 24 hours. It was chosen for its rated tensile strength of up to 5,900 psi. Note that this strength is actually representative of the epoxy itself, not the strength of an epoxy-substrate interface. An applicator gun and 2:1 plunger, shown in Figure 13, was used to keep the mixing ratio as consistent as possible. The exact amount of epoxy used in each test was difficult to keep constant and was not quantifiable. When the epoxy on the stud contacts the substrate, the epoxy is pressed out and forms a chamfer around the edge of the stud diameter. This may have affected the pull-off strength results because the outer edge of the epoxy chamfer is not uniform. Non-uniformity in the stressed area may have contributed to nonlinear responses in the pull-off strength values. Another factor that may have influenced the adhesion results is the mix ratio, which can cause the epoxy to take longer to cure. Even with an

applicator gun and the proper 2:1 plunger, there is no guarantee that the mix ratio was consistently 2:1 in every test.



Figure 13: Loctite E-120HP Epoxy and Applicator Gun

With epoxy on the end of the stud, the actuator is programmed to lower down to the setpoint noted earlier where the stud is just touching the test specimen. Once the stud has made contact, the program is shutdown and the epoxy is left to cure for the desired amount of time. A typical curing position for a test is shown in Figure 14.

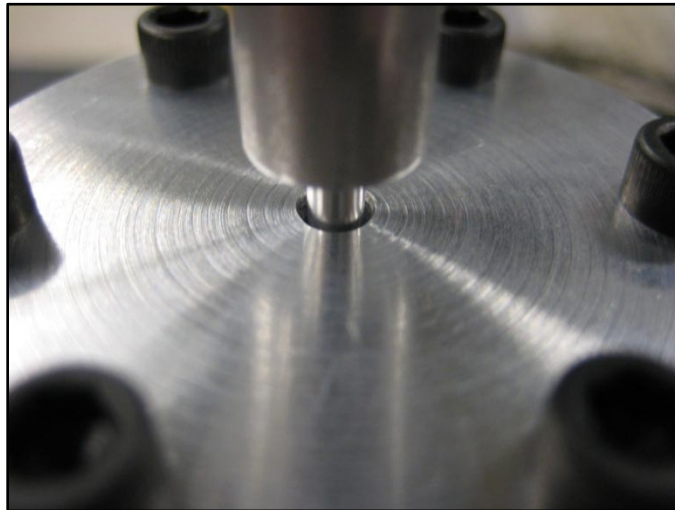


Figure 14: Final Position of Stud during Test Preparation

When the test is ready to be conducted, a program is written that raises the stud at 0.01 mm/s, such that the time to reach maximum stress is no longer than 100 seconds. This rate is chosen based on the 1 MPa/s (150 psi/s) maximum stress rate indicated in the ASTM D4541 standard for pull-off strength testing [14]. Early tests did not use the standard test rate, which may account for inconsistencies in the results. Additionally, dirt accumulation and fingerprints on substrates could have affected adhesion. The aluminum substrates were not carefully cleaned and handled for every test. The stud, however, was thoroughly cleaned with isopropanol to remove the hardened epoxy after every test. The step-by-step process for conducting stud-pull experiments can be found in the Appendix.

4. Experimental Results

Unfortunately, the desired SOFC anode-electrolyte specimens from NexTech Materials were not obtained in time. Instead, epoxy-aluminum and paint-aluminum interfaces were evaluated with stud-pull tests. A total of 36 tests were conducted, of which 29 tests have useful data. Four different interfaces were tested: epoxy to smooth aluminum, epoxy to rough aluminum, epoxy to painted smooth aluminum, and epoxy to painted rough aluminum. Although none of these interfaces have explicit applications to fuel cell interfaces, it is necessary to demonstrate the accuracy and repeatability of the stud-pull instrumentation before testing SOFC specimens. The epoxy-aluminum interface was evaluated primarily to understand how strong the glue is. Several errors and complications were encountered over the 36 tests, some of which resulted in total loss of data and one that severely damaged the test specimen and load cell. If

such an error were to occur while using a real SOFC specimen, the cost of the error would be tremendous.

The first ten tests with useful data were epoxy on approximately 0.050" thick aluminum substrates. Because the stud is also aluminum, these experiments could result in failure at either the stud side or substrate side. When testing first began, the standard separation rate of 0.01 mm/s was not yet implemented: three tests were at 1.0 mm/s, two tests were at 2.0 mm/s, and five tests were at 0.5 mm/s.

4.1. Epoxy-Aluminum Interface Strengths

The rated shear strength of the epoxy on abraded, acid-etched aluminum cured for 12 hours at 65°C is 4800 psi [10]. Figure 15 gives the manufacturer's percent maximum shear strength versus time. The result is a nonlinear function that reaches its maximum after 24 hours.

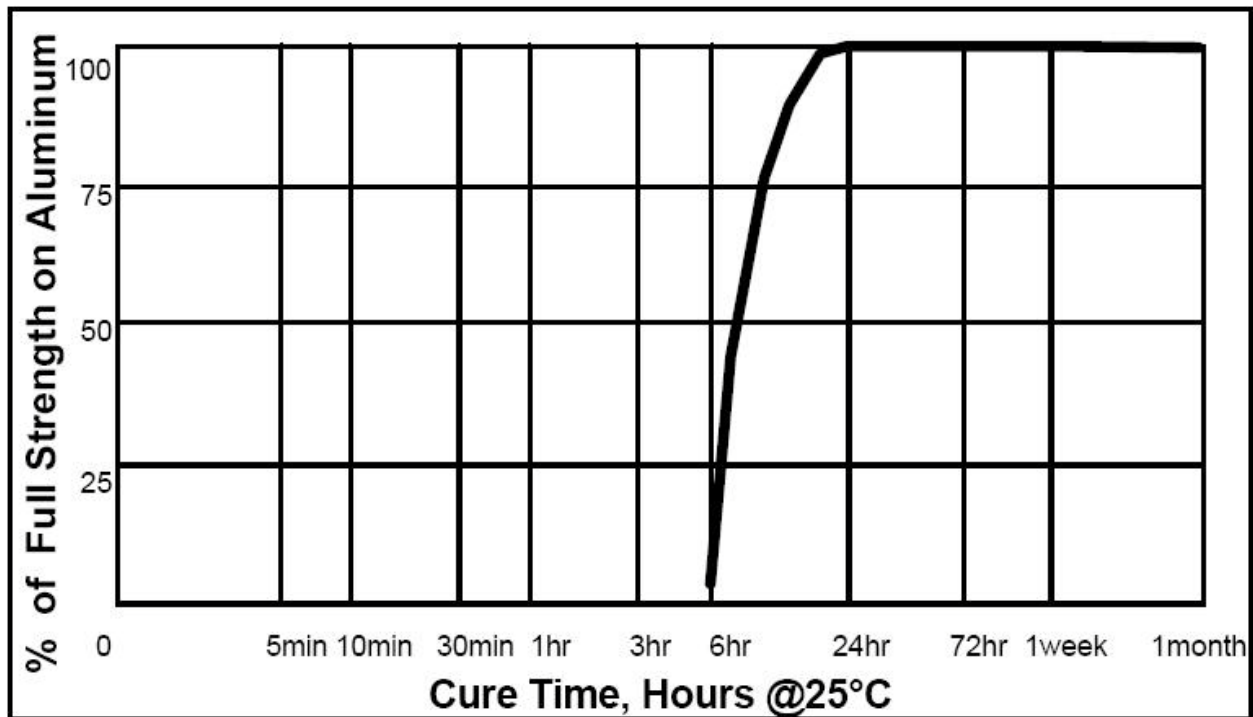


Figure 15: Loctite E-120HP Epoxy Strength vs. Cure Time Curve [10]

Figure 16 shows the *tensile* strength of the epoxy-aluminum interface as a function of cure time in hours. A range for actual strength values was determined by dividing the load at failure by the maximum and minimum stressed areas. The minimum area is the area of the 0.125" diameter stud and the maximum area is the area of the outer diameter of the epoxy after it has been pressed out around the stud-specimen contact point. The maximum strength is load divided by minimum area and the minimum strength is load divided by maximum area. All figures use the minimum stress to characterize pull-off strength. Although the points in Figure 16 represent the tensile strength of the epoxy-aluminum interface, the result should be similar to Figure 15, in that the maximum strength should be level after 24 hours. Instead, the data shows a lack of correlation between strength and cure time, with minimum tensile strength ranging from 180 to 1310 psi with cure time ranging from 18.5 to 167.5 hours. Some of the variables that might corrupt the expected correlation include the high separation rates, the cleanliness of the substrate, the mix ratio of the epoxy, and the substrate surface roughness. The cleanliness of the substrate is not quantifiable, but a dirty substrate would reduce the adhesion strength. The substrate roughness is not identical in every test, so increased degrees of roughness may have an effect on the strength of the epoxy-aluminum interface.

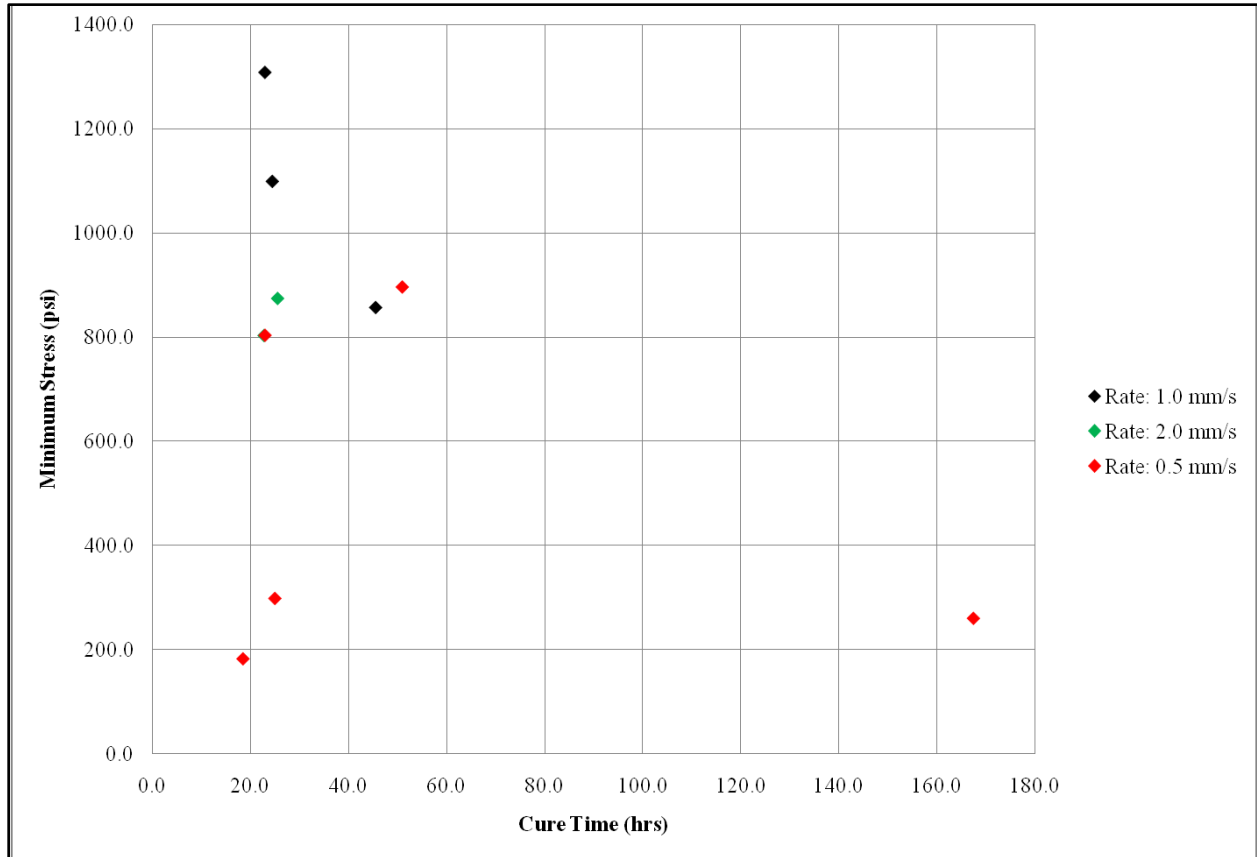


Figure 16: Minimum Stress vs. Cure Time of Epoxy on Smooth Aluminum Substrates

The next seven epoxy-aluminum interface tests were epoxy to sanded aluminum substrates. Except for one, all of these seven experiments were done at 0.01 mm/s. The slower rate and substrate preparation was expected to provide more consistency. The photographic documentation shows that at least one of the seven tests encountered failure at the stud-epoxy interface because there was no residue of epoxy left on the stud after testing. Two other tests showed evidence of failure at the stud-epoxy interface, but the actual location of failure was inconclusive. The results from the seven epoxy-aluminum interface tests at 0.01 mm/s are shown in Figure 17.

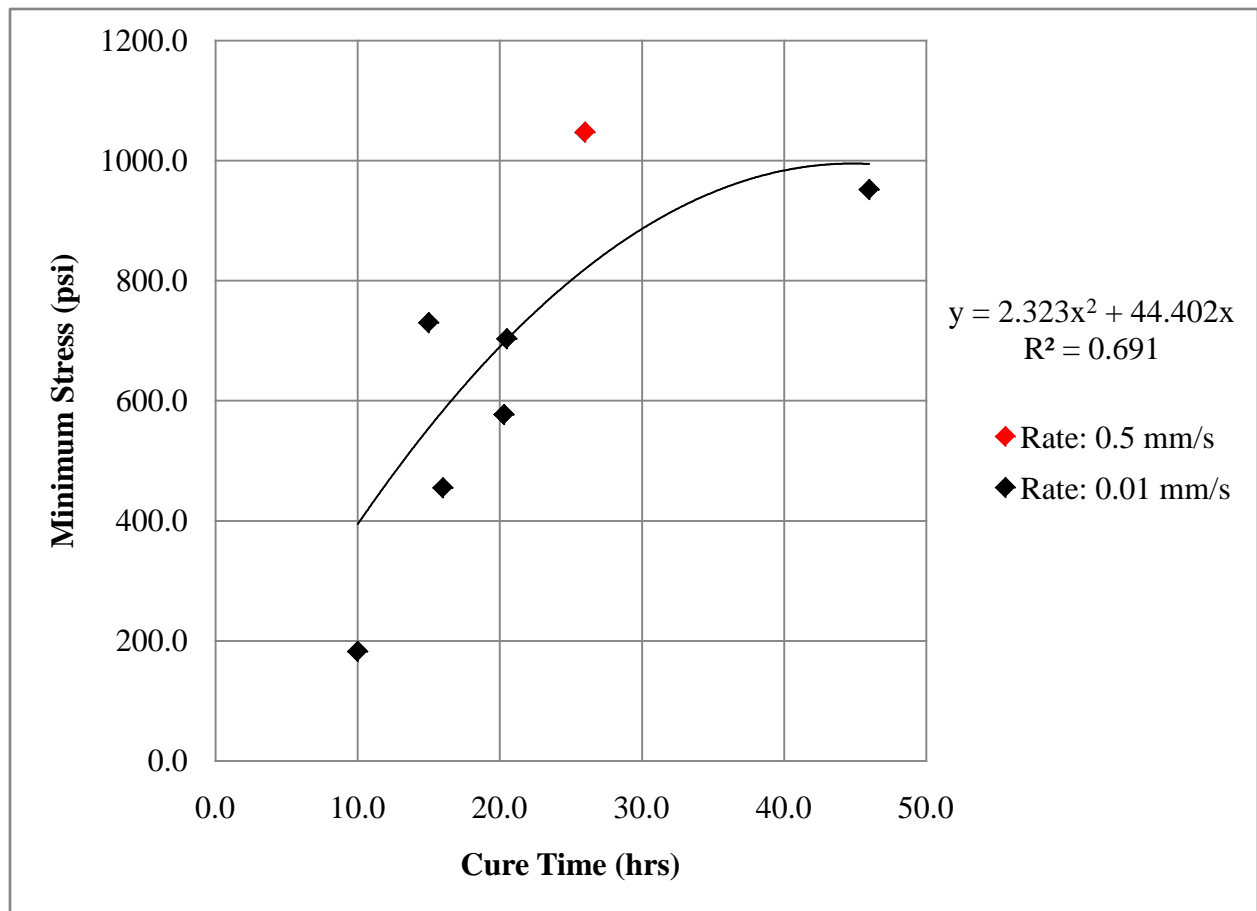


Figure 17: Minimum Stress vs. Cure Time for Epoxy on Rough Aluminum Substrates

The minimum tensile strength varies between about 180 and 1050 psi, but there is evidence of a trend that cure times under 24 hours are less than the maximum strength and cure times over 24 hours are above the maximum strength. If the data is fitted with a second order polynomial trendline with y-intercept set to zero, a curve that begins to resemble the one in Figure 15 can be seen. Assuming a second order trend is applicable, the trendline equation would only be valid for the rising portion of the curve. A longer cure time would not cause the strength of the epoxy to decrease as the equation suggests. Also, the trendline in Figure 17 does not level off until about 45 hours of cure time, but it does demonstrate an expected trend of

increasing strength with increasing cure time. If the epoxy mix ratio is not exactly 2:1 of resin to hardener, the cure time might take longer than 24 hours. Since there is no way to tell retroactively what the mix ratio really was, it is difficult to determine whether the mix ratio plays a critical role in the interface strength.

4.2. Paint-Aluminum Interface Strengths

After the epoxy-aluminum tests were completed, the next set of tests involved a spray-painted aluminum interface. The spray paint was Aervoe Rust Proofing Paint and it was chosen for its availability in the lab. The aluminum substrates were cut to be approximately 0.787" x 0.787" x 0.050" with 0.219" diameter islands of paint. These dimensions coincide with the desired dimensions of a real SOFC specimen with a 20 mm outer diameter and 7/32" diameter islands. An aluminum mask with 7/32" diameter punched holes was built to create the hexagonal pattern of paint islands shown in Figure 18. The islands are larger than the 0.125" stud diameter because the epoxy pushes out to form a larger diameter of approximately 0.200" when it is compressed against the substrate.

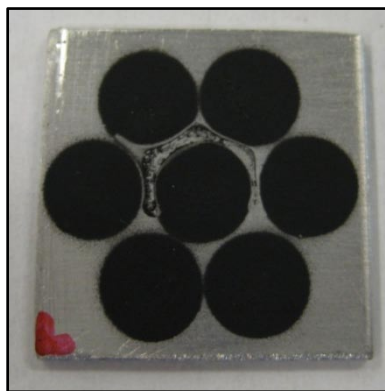


Figure 18: First Painted Aluminum Test Specimen before Testing

The results of the first seven paint tests are shown in Figure 19 and summarized in Table 1. The naming convention in the legend of Figure 19 is in chronological order, where P1-1 is the first test on paint specimen 1, and P1-7 is the seventh test on paint specimen 1. There is no obvious trend from the stress vs. cure time data, which was calculated the same way as before. Three tests conducted with 12 hour cure times experienced significantly different stresses. What is important, however, is that the failure occurred at the coating-substrate interface and not the epoxy-coating interface, as can be seen in Figure 20. The average failure stress on the first painted aluminum test is 426 psi, whereas the average failure stress on the rough aluminum substrate with 0.01 mm/s separation is 600 psi. As long as the epoxy is stronger than the coating-substrate interface, it will give meaningful data about the interface strength.

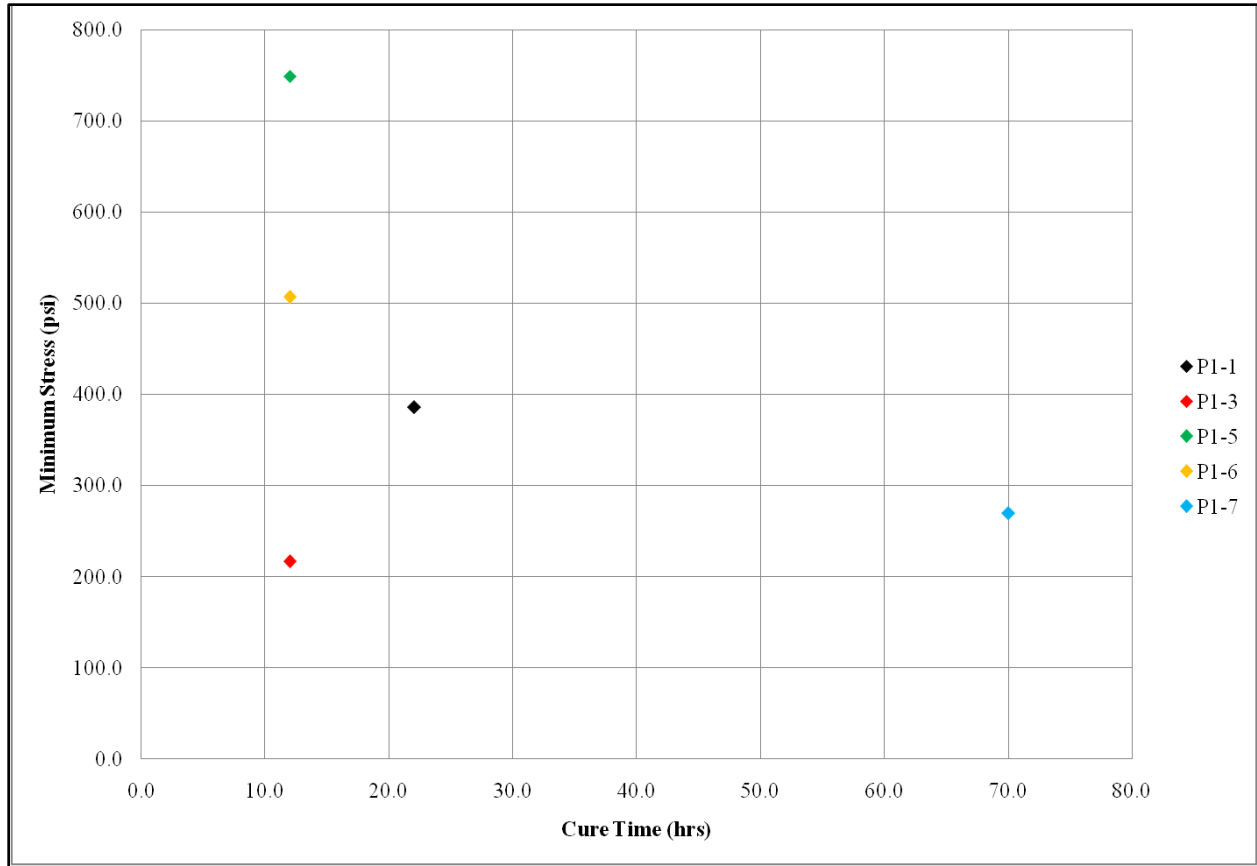


Figure 19: Minimum Stress vs. Cure Time for Epoxy on Painted Smooth Aluminum

Table 1: Summary of Results for 5 Tests on Painted Smooth Aluminum

	Cure Time (hrs)	Peak Load (N)	Minimum Stress (psi)
Mean	25.6	52.2	426
Standard Deviation	25.2	18.9	212
95% Confidence Interval	0.7	0.5	6.0

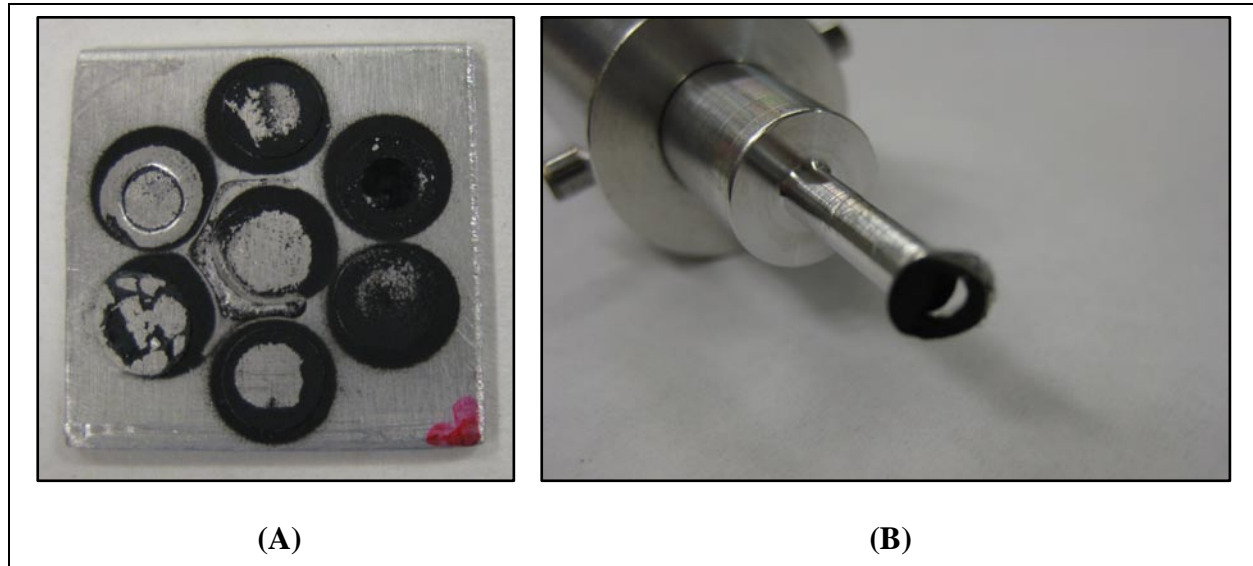


Figure 20: (A) First Painted Aluminum Specimen after Testing and (B) Stud after Testing

The center island in Figure 20A was the first test, the bottom island was the second test, and the rest of the order continues in the clockwise direction. It is clear from the picture that less paint was removed as testing progressed. This may be caused by dirt accumulation on the paint specimen over time or improved paint-aluminum strength over time.

The second painted aluminum specimen had identical dimensions to the first painted specimen, but the surface had been sanded prior to painting. The before-and-after testing views of the specimen can be seen in Figure 21.

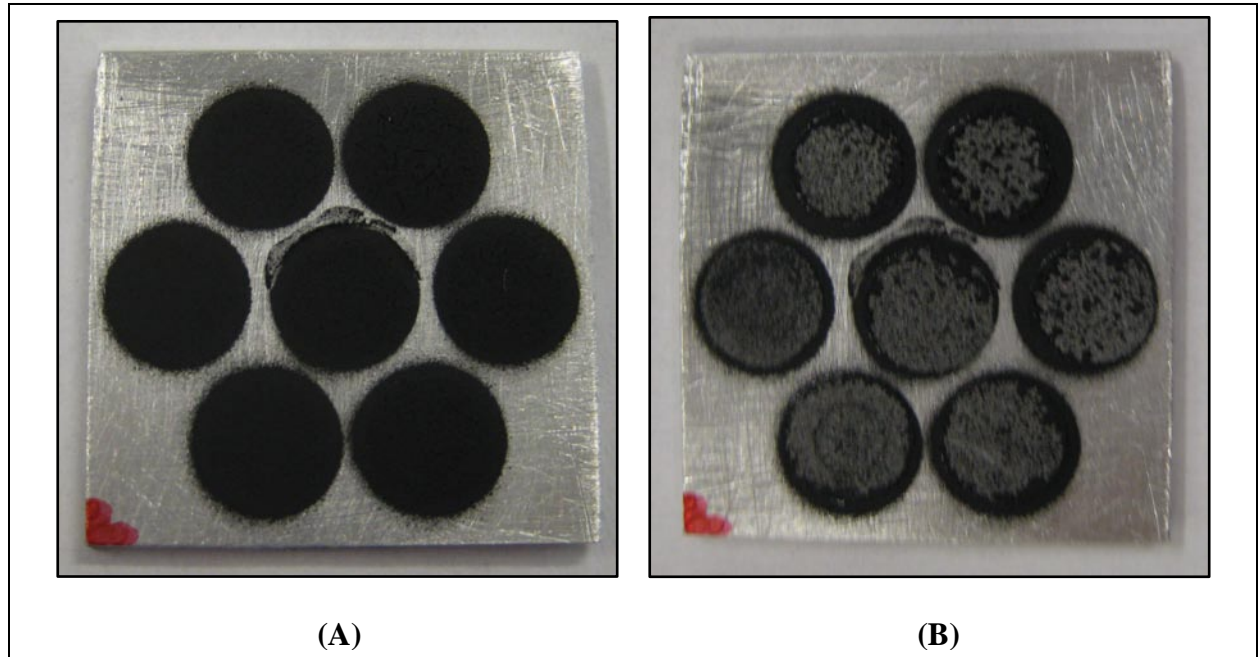


Figure 21: Second Painted Aluminum Specimen: (A) Before Testing and (B) After Testing

The rougher surface still results in failure at the coating-substrate interface, but the paint is not completely removed from the substrate. The paint appears speckled after separation because the paint has filled in the sanded valleys on the surface of the aluminum substrate. The effect of the rougher surface can be seen on the plot of maximum stress vs. cure time for the sanded aluminum specimen shown in Figure 23. A summary of the data statistics is shown in Table 2.

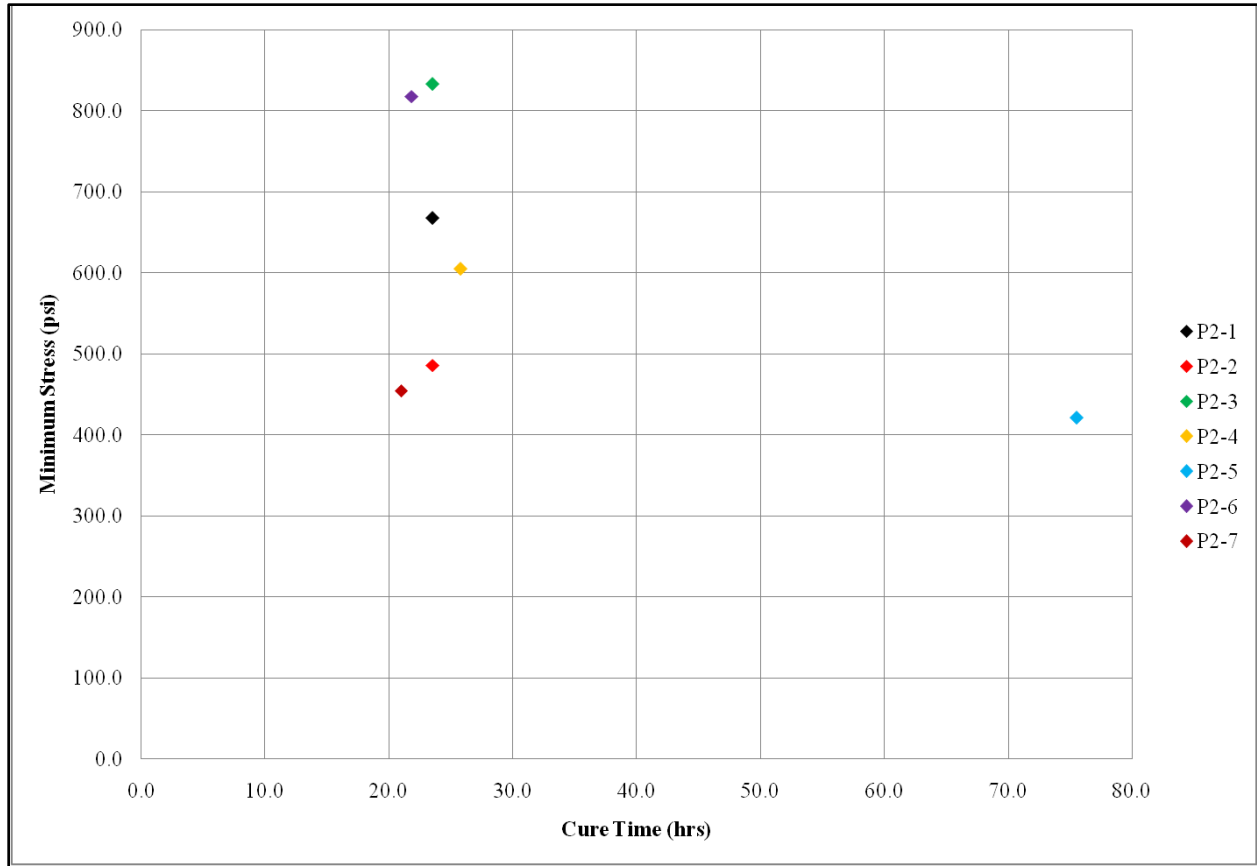


Figure 22: Minimum Stress vs. Cure Time for Epoxy on Painted Rough Aluminum

Table 2: Summary of Results for 7 Tests on Painted Rough Aluminum

	Cure Time (hrs)	Peak Load (N)	Minimum Stress (psi)
Mean	30.7	81.2	612
Standard Deviation	19.8	20.1	169
95% Confidence Interval	0.6	0.6	4.7

The average maximum stress from the seven tests on the painted rough aluminum substrate is 612 psi, which is larger than the 426 psi strength on the painted smooth aluminum substrate. This suggests that increasing the surface roughness on the substrate increases the

strength of the interface. However, the amount of variability in the data shows that more testing should be conducted before the results can be confirmed.

4.3. Other Possible Interface Strength Factors

Other factors besides cure time that were evaluated for having a possible effect on the interface strength are separation rate, preload, mean humidity, and the maximum diameter of the stressed area. Separation rates of 2.0, 1.0, 0.5, and 0.01 mm/s were tested, but there was no apparent correlation between pull-off strength and separation rate. The preload is the initial load on the stud just prior to separating the stud from the specimen. It was measured by the load frame software in real-time and typically ranged between -1.5 N and +0.5 N. Preload data showed that regardless of whether or not the preload was positive or negative, the pull-off strength was not consistent. The mean humidity of the room in which the epoxy is curing is another factor that influences cure time. High relative humidity in the room has been known to weaken epoxy strength [14]. The humidity data was collected from www.wunderground.com for the area code 43210 in Columbus, OH. The humidity of the room in which testing occurs is not necessarily the same as the humidity outside, but the data shows that even if it was the same, there is no consistent correlation. Finally, the max epoxy diameter was measured using a digital caliper around the outer edge of the epoxy that has pushed out after compressing against the test specimen. The minimum stress is calculated by dividing the peak load by the maximum stressed area. It is difficult to determine exactly what the stressed area is in each experiment because the location of failure is not always consistent. The majority of measured diameters are within ± 0.025 in of 0.200" outer diameter, but the stress ranges from below 200 psi to above 1300 psi without any apparent trends.

4.4. Load-Displacement Results

The main data recorded by the load frame software for which all of the other data is derived is the load vs. displacement. Plots for load vs. displacement of the 0.01 mm/s separation rate tests of epoxy-aluminum are shown in Figure 23.

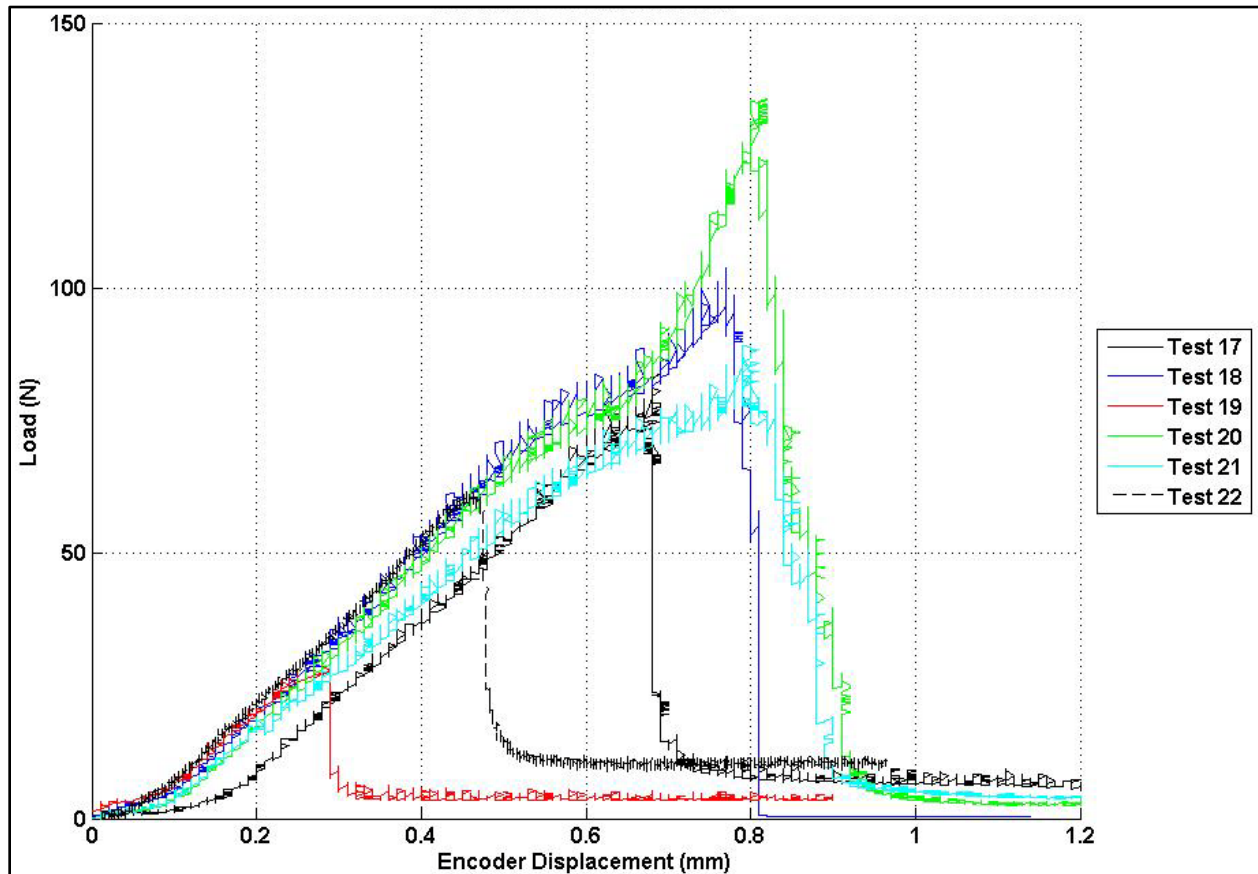


Figure 23: Load vs. Displacement of 0.01 mm/s Tests of Epoxy to Rough Aluminum

The shape of most of the curves is generally nonlinear for the first 0.1 mm, linear from 0.1 to 0.5 mm, nonlinear from 0.5 mm to 0.7 mm, and again linear until failure. A similar trend is apparent in the load vs. displacement curves of the painted specimens, shown in Figure 24.

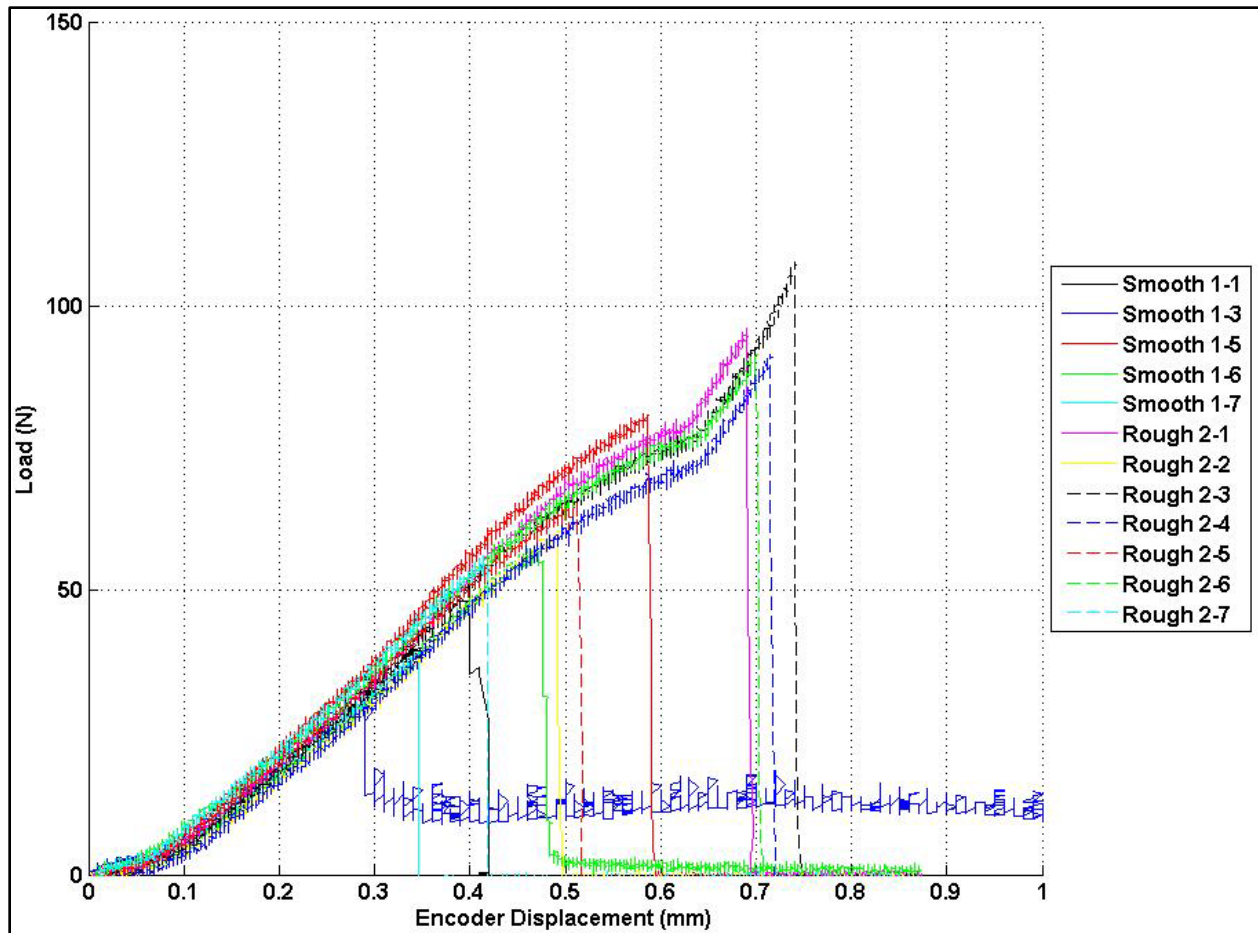


Figure 24: Load vs. Displacement of 0.01 mm/s Tests of Epoxy to Painted Aluminum

The curves in Figure 24 are generally nonlinear for the first 0.1 mm, linear from 0.1 to 0.45 mm, nonlinear from 0.45 to 0.65 mm, and again linear until failure. These trends seem to occur even if the surface is smooth, although only the rougher surfaces experienced failure after more than 0.6 mm of displacement. The consistently linear curves for all interfaces show an elastic response of load to displacement, at least for the first 0.5 mm of separation. It is not known at this time what causes the increased slope after 0.65 mm of displacement.

5. Conclusions and Future Work

The main accomplishment of this project was designing, building, and troubleshooting an experimental apparatus and becoming familiar with a load frame and its data acquisition software. It is hardly a trivial process to design a stud-pull tester and use it enough times to get repeatable results under consistent operating conditions. Even though the SOFC specimens that were the motivation for the project never arrived, valuable and necessary baseline data was collected. The results are summarized in Table 3 and Table 4 (all test data can be found in the Appendix). On average, the epoxy-aluminum interface is stronger than the paint-aluminum interface and the painted rough aluminum interface is stronger than the painted smooth aluminum interface. Based on the amount of variability within the small sample sizes, additional testing would have to be completed to determine conclusively whether or not these trends are actually valid, but this requires a significant amount of time. While the current data shows that there is no correlation between pull-off strength and preload, separation rate, humidity, or stressed area, there are some additional variables that might be significant. At a rate of one test per day, a full design of experiments that evaluates different interfaces against additional variables such as surface roughness, substrate cleanliness, and deposition temperature would take several weeks or months.

Table 3: Summary of Average Data for All Tests

Rate (mm/s)	Mean Peak Load (N)	Minimum Stress (psi)	Samples
0.01 - Paint, Smooth	52.2	426	5
0.5	80.2	582	6
0.01	84.2	600	6
0.01 - Paint, Rough	81.2	612	7
2	108.7	839	2
1	123.4	1088	3

Table 4: Summary of Painted Specimen vs. Non-Painted Specimen Data

Test Type	Mean Peak Load (N)	Minimum Stress (psi)	Samples
Paint-Substrate	69.1	534	12
Glue-Substrate	92.6	708	17

The current stud-pull apparatus has several features that can be improved for future testing. First, the tests that were conducted introduce a degree of bending in the specimen because of the washer between the specimen and the retaining ring. The washer creates a necessary gap between the retaining ring and the specimen to allow for epoxy overflow during test preparation, but the washer holds the edges of the test specimen down while the stud pulls on the center of the specimen. A purely tensile method would have the diameter of the coating area exactly equal to the diameter of the stud and the base. As long as the base, stud, and test piece are aligned, the test would measure purely tensile force.

The second challenge is to improve the efficiency of running experiments. Under the present conditions, the load cell side of the apparatus and the fixed base of the apparatus become one rigid piece once the stud and epoxy contact the test specimen. There is no way to effectively remove the stud from the load cell without compromising the experiment until 24 hours later when the testing is ready to be done. A more time-efficient method might be to have a detachable stud that can be separated from the load cell once the epoxy has had about 2 hours to set. If the load cell could be removed, the base plate could be unscrewed from the load frame base and the setup could be set aside to cure for the remaining 22 hours. Meanwhile, an identical setup with a base plate, table, retaining ring, and detachable stud could be put into the load frame and another test could be prepared. The drawbacks to this method are that consecutive tests

could not be performed on the same specimen and there is no way to measure the compressive preload on the test specimen once it is removed from the load cell.

If SOFC specimens do come in from NexTech Materials, they will look similar to the painted specimens. A solid model of an anode-electrolyte specimen made of a nickel oxide gadolinium doped ceria and nickel oxide yttria stabilized zirconia composite (NiO-GDC/NiO-YSZ) anode and yttria stabilized zirconia (YSZ) electrolyte is shown in Figure 25. The electrolyte substrate is 20 mm diameter and 150 μm thick, while the anode islands are 3/16" diameter and 750 μm thick.

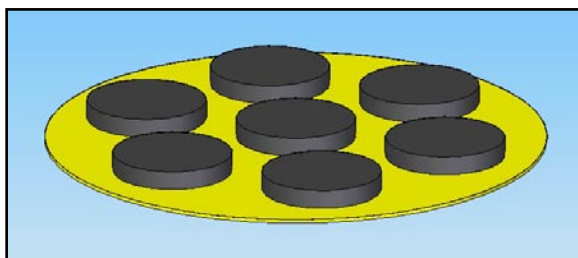


Figure 25: Model of NiO-GDC/NiO-YSZ Anode-Electrolyte Test Specimen from NexTech

The first tests on the specimens would be as-received, meaning the specimen would go through stud-pull testing without any variation in the material. Once a baseline of data is collected, the specimen would be reduced with a forming gas of nitrogen and hydrogen. This reduction is necessary to remove the oxygen from the NiO-YSZ anode so that the anode can conduct electrons. The result of the reduction would yield a small amount of water and Ni-YSZ, which is a good electron conductor. After reduction, the specimen would be tested to determine what effect the reduction has on the interface strength of the specimen. Additional testing could be done on thermally cycled specimens to evaluate what effect heating and cooling have on interface strength.

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Appendix

Tom Knapp
Thursday 4/24/2008

Operation of load frame:

All stud-pull tests were conducted using an 800LE3 series load frame from TestResources. The accompanying software features real-time displays of the load and position of the load frame actuator. The software also enables the user to program the actuator to follow specific motions and record the data. Upon completion of each test, the data can be exported into a Microsoft Excel spreadsheet for analysis.

Step-by-step instructions for a stud-pull test:

1. Turn on the power strip
2. Turn on the computer
3. Turn knob on power pack from OFF to ON and toggle the Fault Reset switch
4. Turn on the controller
5. Log in to computer (username: Operator, password: lannutti)
6. From the desktop, go to My Computer/C: Drive/Program Files/MTL-Windows/Data
7. Make a new folder and rename it in the format YYYY-MM-DD-LASTNAME (e.g. 2008-04-24-Knapp)
8. From the desktop, open software OSU 800L
9. Wait approximately 10 seconds for real-time display to open
10. If the default file that loads is not the desired file, load the desired file or save it as a new one
11. At the bottom of the Console window, change the Control Mode to the desired position (Encoder)
12. Check that the Limit Interlocks are specified at safe values
13. If a new test is being prepared, see Stud Pull-Test Preparation
14. If a new test is being conducted, see Stud Pull-Testing

Stud-Pull Test Preparation:

1. Mount the table to the base plate
3. Place the test piece in the desired position on the table (centered appropriately)
4. Place the retaining cylinder (washer) over the test piece (centered appropriately)
5. Place the retaining ring over the washer (centered appropriately)
6. Use fingers to center the retaining ring on the table using the six screws (do not tighten)
7. Place the jig over the table to check alignment
8. If alignment is good, use fingers turn the screws until they are just touching the retaining ring (do not tighten)
9. At this point, the ring should not move unless the screws are undone
10. Mount the base plate to the load frame
11. On the load cell side, screw the ball joint to the load cell
12. Screw the stud holder to the ball joint
13. Pin the stud into the stud holder (the stud and stud holder may already be together)

14. Roughly center the stud over the retaining ring
15. Using Encoder control, lower the stud to just above the retaining ring center hole
16. Center the stud to the retaining ring center hole
17. Use the MTL-Programming window to program the stud to very slowly drop down to the test surface (0.01 mm/s)
18. Once the stud touches the test surface, compressive force will increase
18. Make sure the position at this point does not result in more than about 1.0 N of compressive force
19. If the stud is still centered, raise it back up to an appropriate level so epoxy can be applied
20. Prepare all documentation and preload code before applying epoxy to the stud
21. If everything is ready for testing, mix the epoxy for 1-2 minutes
22. When the epoxy is thoroughly mixed, apply a dab to the bottom end of the stud (be careful not to apply too much)
23. Use the MTL-Programming window to run the code that slowly drops the stud to the appropriate level
24. Note all of the control settings and the preparation time
25. In the Console tab, save the file with an appropriate name (e.g. 2008-04-24-Test4Prep)

Stud-Pull Testing

1. Before testing, go to the Console tab and save the file with an appropriate name (e.g. 2008-04-24-Test4)
2. Note all of the control settings and the curing time
3. If the epoxy has cured for the desired amount of time, go to the MTL-Programming tab
4. On the right side, under Recording, turn on Auto On/Off (this will automatically record data after tests)
5. Program the stud to rise at 0.01 mm/s for ~1.5 mm and then rise at 2 mm/s for ~30 mm
6. Save the code with an appropriate name (e.g. 2008-04-24-Test4)
7. When the test is finished and the set point has been reached, go DIRECTLY to the Data Acquisition tab
8. Click Export
9. In the new window, choose Export in the dropdown menu and click Data (this generates an Excel file)
10. In the folder where files are saving, make sure that the Excel file is there
11. If there is no Excel file, go DIRECTLY back to the MTL-Programming tab and click Stop under MTL-Code Control
12. Export data using the previous instructions (if instructions are not followed exactly, data can be easily lost)
13. Once data has been exported properly, document the results of the tests and prepare for a new test
14. Use Isopropanol on a paper towel to remove excess epoxy from stud

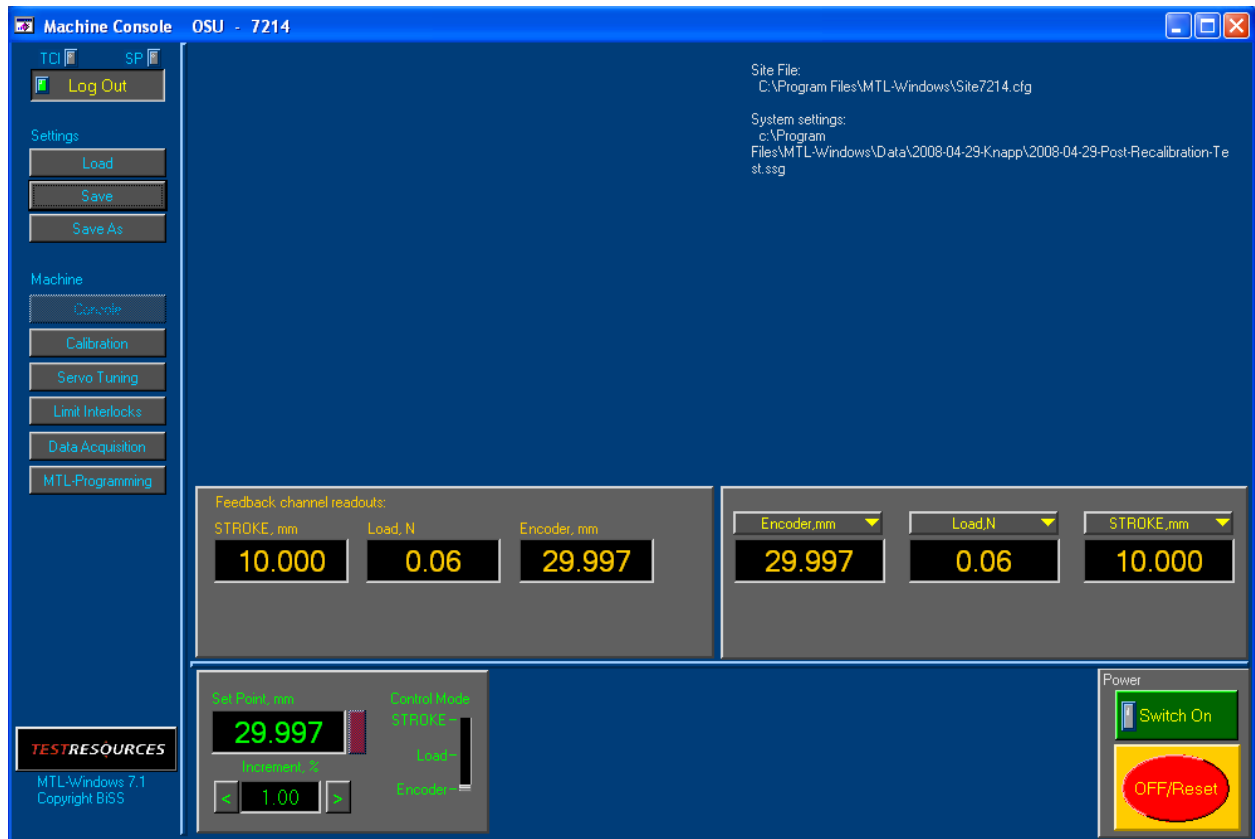


Figure 26: TestResources Software Console Window Real-Time Display

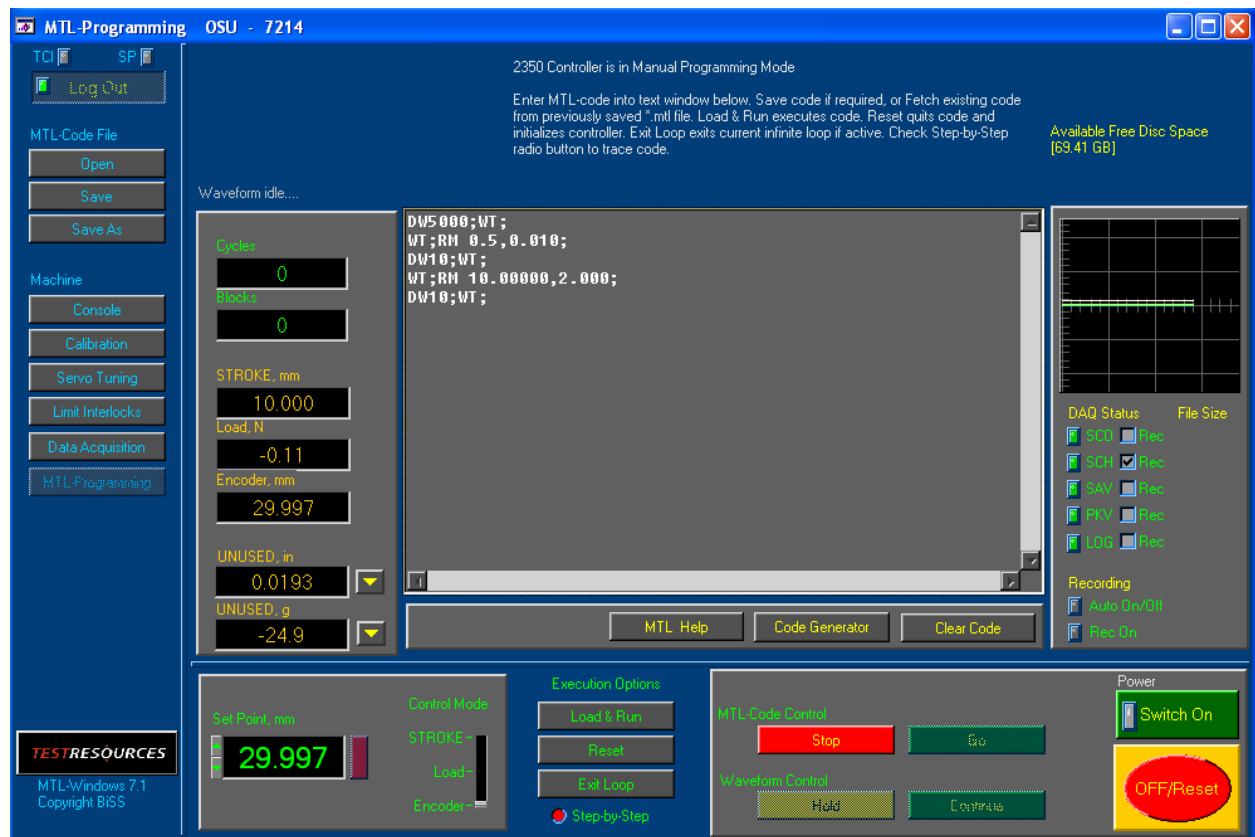


Figure 27: TestResources Software MTL-Programming Window Real-Time Display

Table 5: All Test Data

Test #	Cure (hrs)	Rate (mm/s)	Disp (mm)	dmin (in)	dmax (in)	Max Load (N)	Preload (N)	Mean Humidity	Interface Description
4	23.0	1	0.81	0.125	0.185	156.4	-0.77	61	Epoxy, smooth
5	45.5	1	0.59	0.125	0.183	100.3	-0.81	70	Epoxy, smooth
6	24.5	1	0.60	0.125	0.172	113.6	-0.47	74	Epoxy, smooth
9	22.8	2	0.54	0.125	0.183	94.0	-0.38	83	Epoxy, smooth
10	25.5	2	0.71	0.125	0.201	123.4	-0.48	68	Epoxy, smooth
11	51.0	0.5	0.70	0.125	0.193	116.6	-0.84	61	Epoxy, smooth
12	23.0	0.5	0.65	0.125	0.201	113.4	1.46	62	Epoxy, smooth
13	18.5	0.5	0.23	0.125	0.200	25.6	0.08	56	Epoxy, smooth
14	25.0	0.5	0.32	0.125	0.191	38.1	-0.34	56	Epoxy, smooth
15	167.5	0.5	0.35	0.125	0.196	35.0	-0.43	51	Epoxy, smooth
16	26.0	0.5	0.84	0.125	0.204	152.3	0.55	44	Epoxy, rough
17	20.5	0.01	0.69	0.125	0.185	84.1	0.12	46	Epoxy, rough
18	20.3	0.01	0.77	0.125	0.227	103.9	0.27	51	Epoxy, rough
19	10.0	0.01	0.29	0.125	0.218	30.3	0.85	51	Epoxy, rough
20	46.0	0.01	0.82	0.125	0.202	135.7	-0.34	71	Epoxy, rough
21	15.0	0.01	0.79	0.125	0.187	89.2	-0.37	64	Epoxy, rough
22	16.0	0.01	0.463	0.125	0.197	61.7	-1.00	55	Epoxy, rough
P1-1	22.0	0.01	0.40	0.125	0.195	51.3	-0.34	61	Paint, smooth
P1-3	12.0	0.01	0.29	0.125	0.206	32.2	0.00	55	Paint, smooth
P1-5	12.0	0.01	0.588	0.125	0.176	81.0	-0.50	43	Paint, smooth
P1-6	12.0	0.01	0.46	0.125	0.180	57.4	-1.00	60	Paint, smooth
P1-7	70.0	0.01	0.346	0.125	0.204	39.2	-1.25	58	Paint, smooth
P2-1	23.5	0.01	0.69	0.125	0.203	96.1	-0.75	48	Paint, rough
P2-2	23.5	0.01	0.492	0.125	0.189	60.6	-1.00	63	Paint, rough
P2-3	23.5	0.01	0.742	0.125	0.192	107.3	-1.00	89	Paint, rough
P2-4	25.8	0.01	0.715	0.125	0.208	91.4	-1.50	80	Paint, rough
P2-5	75.5	0.01	0.508	0.125	0.211	65.5	-1.75	74	Paint, rough
P2-6	21.8	0.01	0.699	0.125	0.179	91.5	-1.00	69	Paint, rough
P2-7	21.0	0.01	0.414	0.125	0.188	56.1	-1.25	76	Paint, rough

LOCTITE

1001 Trout Brook Crossing
Rocky Hill, CT 06067-3910
Telephone: (860) 571-5100
FAX: (860) 571-5465

Technical Data Sheet

Hysol® Product E-120HP

formerly Durabond E-120HP
Industrial Version, August 2001

PRODUCT DESCRIPTION

LOCTITE® Hysol® Product E-120HP is a high-viscosity, non-sagging industrial grade epoxy adhesive with extended work life. Once mixed, the two-component epoxy cures at room temperature to form a tough, amber-beige bondline with excellent resistance to peel & impact forces. When fully cured, the epoxy offers superior thermal shock resistance, excellent mechanical and electrical resistant properties, and withstands exposure to a wide variety of solvents and chemicals.

TYPICAL APPLICATIONS

Originally designed for bonding nose cones in aerospace applications. Also suitable for low stress, general industrial applications which require high impact and high peel strength. Bonds dissimilar materials including aluminum, steel, and other metals, as well as a variety of plastics & ceramics.

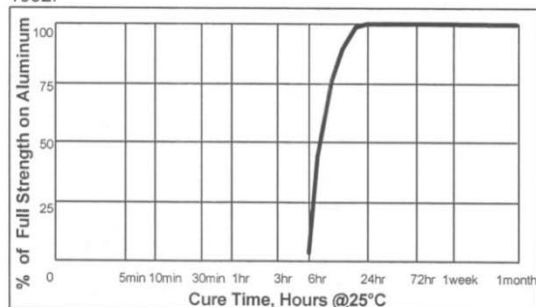
PROPERTIES OF UNCURED MATERIAL

Resin	Value	Typical Range
Chemical Type	Epoxy	
Appearance	Pale yellow liquid	
Specific Gravity @ 25°C	1.10	1.0 to 1.2
Viscosity @ 25°C, mPa.s (cP)	41,500	31,000 to 51,000
Flash Point (TCC), °C (°F)	>93 (>200)	
Hardener	Value	Typical Range
Chemical Type	Polyamide	
Appearance	Light colored liquid	
Specific Gravity @ 25°C	1.00	0.9 to 1.1
Viscosity @ 25°C, mPa.s (cP)	2,800	2,000 to 4,000
Flash Point (TCC), °C (°F)	160 (320)	
Mixture	Typical Value	
Appearance	Amber-Beige	
Specific Gravity @ 25°C	1.07	
Mix Ratio (R:H) by Weight	100 to 46	
by Volume	2 to 1	

TYPICAL CURING PERFORMANCE

Cure speed

The graph below shows the shear strength developed over time on abraded, acid etched aluminum lap shears with an average bondline gap of 3 to 9 mils and tested according to ASTM D-1002.



Curing Properties

(@ 25°C unless noted)	Typical Value
Working Life, minutes	120
Tack Free time (low humidity), minutes	140

TYPICAL PROPERTIES OF CURED MATERIAL

(@ 25°C unless noted)	Typical Value
Physical Properties	
Dielectric Strength, Volts/Mil	650
Tensile Strength ASTM D638, psi	5,910
Tensile Elongation ASTM D-638, %	10
Hardness ASTM D-1706, Shore D	85
Glass Transition Temperature, Tg, °C	90

PERFORMANCE OF CURED MATERIAL

Shear Strength vs Substrate

(Substrates cured for 12 hours @ 65°C with no induced gap.)

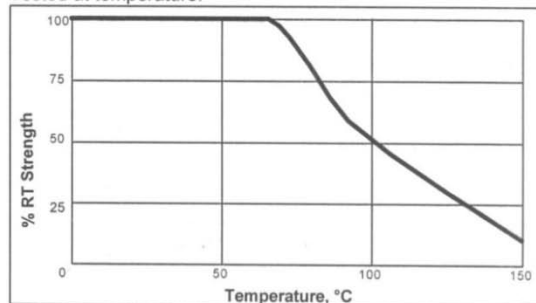
Substrate	Typical Value
Lap Shear	N/mm ² (psi)
Grit-Blasted Steel	29.4 4270
Aluminum (Abraded/Acid Etched 3 to 9 mil gap)	33.1 4800
Aluminum (Anodized)	14.6 2120
Stainless Steel	23.1 3350
Polycarbonate	7.0 1010
Nylon	2.3 330
Wood (Fir)	11.3 1640
Block Shear	N/mm ² (psi)
PVC	11.7 1700
ABS	7.5 1090
Epoxy	20.1 2920
Acrylic	1.5 220
Glass	22.7 3290

TYPICAL ENVIRONMENTAL RESISTANCE

Hot Strength

Test procedure :	ASTM D-1002
Substrate:	Abraded, acid etched aluminum
Bondline gap, mils:	3 to 9
Cure procedure:	12 hours at 65°C & 4 hours at 22°C

Tested at temperature.

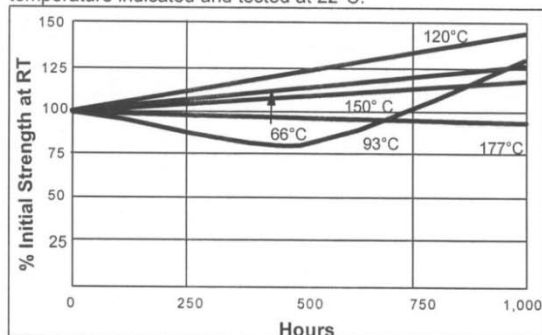


NOT FOR PRODUCT SPECIFICATIONS.
THE TECHNICAL DATA CONTAINED HEREIN ARE INTENDED AS REFERENCE ONLY.
PLEASE CONTACT LOCTITE CORPORATION QUALITY DEPARTMENT FOR ASSISTANCE AND RECOMMENDATIONS ON SPECIFICATIONS FOR THIS PRODUCT.
ROCKY HILL, CT FAX: +1 (860)-571-5473 DUBLIN, IRELAND FAX: +353-(1)-451-9959

Figure 28: Loctite E-120HP Epoxy Data Sheet 1 [10]

Heat Aging

Cured for 5 days at 22°C on steel with no induced gap, aged at temperature indicated and tested at 22°C.

**Chemical / Solvent Resistance**

Cured for 5 days at 22°C on steel with no induced gap, aged under conditions indicated and tested at 22°C.

Solvent	Temp.	% Initial Strength retained at	
		500 hr	1000 hr
Air	87°C	-	98
Motor Oil (10W-30)	87°C	126	119
Unleaded Gasoline	87°C	-	105
Water/Glycol (50%/50%)	87°C	91	89
Salt/Fog ASTM B-117	22°C	-	44
95% Relative Humidity	38°C	-	78
Condensing Humidity	49°C	-	60
Water	22°C	-	72
Acetone	22°C	-	99
Isopropyl Alcohol	22°C	-	108

GENERAL INFORMATION

This product is not recommended for use in pure oxygen and/or oxygen rich systems and should not be selected as a sealant for chlorine or other strong oxidizing materials.

For safe handling information on this product, consult the Material Safety Data Sheet, (MSDS).

Directions for use

- For high strength structural bonds, removal of surface contaminants such as paint, oxide films, oils, dust, mold release agents and all other surface contaminants.
- Use gloves to minimize skin contact. DO NOT use solvents for cleaning hands.
- Dual Cartridges:** To use simply insert the cartridge into the application gun and start the plunger into the cylinders using light pressure on the trigger. Next, remove the cartridge cap and expel a small amount of adhesive to be sure both sides are flowing evenly and freely. If automatic mixing of resin and hardener is desired, attach the mixing nozzle to the end of the cartridge and begin dispensing the adhesive. For hand mixing, expel the desired amount of the adhesive and mix thoroughly. Mix approximately 15 seconds after uniform color is obtained. **Bulk Containers:** Mix thoroughly by weight or volume in the proportions specified in Properties of Uncured Material section. Mix vigorously approximately 15 seconds after uniform color is obtained.

- For maximum bond strength apply adhesive evenly to both surfaces to be joined.
- Application to the substrates should be made within 2 hours. Larger quantities and/or higher temperatures will reduce this working time.
- Join the adhesive coated surfaces and allow to cure at 25°C (77°F) for 24 hours for high strength. Heat up to 93°C (200°F), will speed curing.
- Keep parts from moving during cure. Contact pressure is necessary. Maximum shear strength is obtained with a 3-9 mil bond line.
- Excess uncured adhesive can be cleaned up with ketone type solvents.

Storage

Product shall be ideally stored in a cool, dry location in unopened containers at a temperature between 8°C to 28°C (46°F to 82°F) unless otherwise labeled. Optimal storage is at the lower half of this temperature range. To prevent contamination of unused product, do not return any material to its original container. For further specific shelf life information, contact your local Technical Service Center.

Data Ranges

The data contained herein may be reported as a typical value and/or range. Values are based on actual test data and are verified on a periodic basis.

Note

The data contained herein are furnished for information only and are believed to be reliable. We cannot assume responsibility for the results obtained by others over whose methods we have no control. It is the user's responsibility to determine suitability for the user's purpose of any production methods mentioned herein and to adopt such precautions as may be advisable for the protection of property and of persons against any hazards that may be involved in the handling and use thereof. In light of the foregoing, **Loctite Corporation specifically disclaims all warranties expressed or implied, including warranties of merchantability or fitness for a particular purpose, arising from sale or use of Loctite Corporation's products. Loctite Corporation specifically disclaims any liability for consequential or incidental damages of any kind, including lost profits.** The discussion herein of various processes or compositions is not to be interpreted as representation that they are free from domination of patents owned by others or as a license under any Loctite Corporation patents that may cover such processes or compositions. We recommend that each prospective user test his proposed application before repetitive use, using this data as a guide. This product may be covered by one or more United States or foreign patents or patent applications.

Figure 29: Loctite E-120HP Epoxy Data Sheet 2 [10]